METHOD FOR THE MEASUREMENT OF COLOUR OF RAW WOOL

0. Brief History

This method has been prepared by the IWTO Technology & Standards Committee. It is the result of close collaboration between a number of organisations in Australia and New Zealand (viz. AWTA Ltd., CSIRO, UNSW, AWC, WRONZ, NZWTA, SGS) to arrive at an agreed procedure to cover the specific requirements and differing wool types of those 2 countries. In particular it was essential that it be compatible with existing or planned national standards.

The first national standard was NZS 8707-1977[1], issued by the Standards Association of New Zealand in December 1977, which was restricted in application to commercially scoured wool. Subsequently this method was amended and extended to cover all raw wool in New Zealand in the form of NZS 8707-1984, issued in July 1984. The development of these 2 New Zealand Standards resulted from investigations carried out by the Wool Research Organisation of New Zealand. Following the 1984 issue, further development of the method for commercial test house use has been carried out by the New Zealand Wool Testing Authority.

In Australia, work since 1977 has resulted in the development of AS3546[2] for the measurement of average yellowness for base colour in greasy wool and average yellowness for base and "as is" colour of wool in scoured or carbonised form.

In 1986 a document, which represented a combination of both the Australian and New Zealand Standards was presented to IWTO for endorsement as a Draft Test Method. The current text incorporates all amendments approved up to and including the IWTO Meeting, Nice, November 2002.

1. Foreword

It is difficult for the human eye to make reliable quantitative assessments of colour[3] and there can be considerable variability in the degree of accuracy with which observers detect differences in colours. Moreover, colour differences, which are easy to see when the objects are side-by-side, are very much harder to assess when the objects are separated by time or distance. An objective measurement of colour is therefore desirable.

A complete measurement of the colour of an opaque material such as wool is obtained by measuring the proportion of light reflected from its surface throughout the visible spectrum. It is, however, possible to obtain useful information by measuring the reflection in the red, green and blue regions. Under the conditions specified by the International Commission of Illumination, (CIE)[4], these 3 readings are referred to as the tristimulus values X (red), Y (green) and Z (blue). The values provide sufficient information to describe colour for the specified light source and viewing conditions. They can be used directly, or they can be transformed, to provide information concerning particular aspects of colour.

The colour of wool is influenced, not only by its inherent colour, but also by the grease, suint, dirt, vegetable matter and other impurities present. The colour of the cleaned wool is usually of interest since most of these impurities are removed during processing and generally do not affect the colour of the final product. This Test Method relates to a measurement on either a laboratory sample prepared by the carding method in accordance with Appendix B of this Specification. For greasy wool, a core sample is scoured and carded to remove as much of these contaminants as possible, so as to achieve base colour.

The base colour measurement of either greasy wool core samples or commercially scoured wool is an attempt to determine the inherent colour of the wool. The “as is” colour of commercially scoured or carbonised wool, when compared with its base colour, is an indicator of the effectiveness of commercial scouring.

The Yellowness Index given by this Specification is derived from the tristimulus values Y and Z as Y-Z. As an illustration of the range of this parameter for wool, the following values are given and compared with typical values that might have been obtained with the earlier version of this Test Method that used the C/2 colour space:

Colour Description	Y-Z Units	
	D65/ 10^{o}	C/2^{o}
Very White	7	-2
White	8	0
Creamy	11	3
Canary Yellow Stain	18	12

Wool colours, which are not well identified solely by yellowness measurement, include brown, black and green.

2. Scope

2.1 This Specification sets out a method for measuring base colour of greasy wool and base and “as is” colour of commercially scoured or carbonised wool samples taken from bales according to the relevant IWTO Regulations. Procedures for the preparation of the wool before measurement are described.

2.2 The method is not suitable for detecting the presence of individual coloured fibres.

3. Principle

3.1 A representative sample of wool is obtained by coring.

3.2 The preparation of test specimens for colour measurement includes subsampling, scouring (only if base colour is required), removal of vegetable matter contamination, drying and conditioning in the standard laboratory atmosphere as specified in IWTO-52. All these processes should be carried out in a manner which minimises modification of the base colour of the test specimen.

3.3 For “as is” tests for commercially scoured or carbonised wool, a representative sample should be drawn from the internal parts of packages or consignments to avoid parts of the sample being accidentally soiled.

3.4 The prepared test specimen is measured to give the tristimulus values X, Y, and Z for the CIE* Illuminant D65 and 10^{o} Observer. Between-instrument variations are reduced by the use of certified tiles to calibrate the instrument.

* CIE - Commission Internationale de L’Eclairage

4. Definitions

4.1 Raw Wool

Greasy wool; wool which has been scoured, carbonised, washed or solvent-degreased; scoured skin wools; and slipe wools. It consists of wool fibres together with variable amounts of vegetable matter and extraneous alkali-insoluble impurities, mineral matter, wool waxes, suint and moisture.

4.2 Greasy Wool

Wool, from the sheep's back or sheepskins, which has not been scoured, solvent-degreased, carbonised or otherwise processed.

4.3 Sample

4.3.1 Core Sample

A representative sample of wool obtained by coring. (When issuing an IWTO Certificate, sampling must comply with the IWTO Core Test Regulations or the IWTO Condition Testing Regulations for Scoured or Carbonised Wool.)

4.3.2 Subsample

The randomly drawn portion, representative of the sample. For base colour measurement of cored raw wool samples, the subsamples are to be individually scoured and dried.

4.3.3 Laboratory Sample

The randomly drawn carded sample, representative of all the subsamples, prepared for measurement.

4.3.4 Test Specimen

The conditioned, randomised portion, representative of the laboratory sample, to be measured.

4.4 Brightness

For raw wool a measure of the intensity of reflectance expressed as the Y tristimulus value.

4.5 Yellowness

For raw wool the difference between the Y tristimulus value and the Z tristimulus value (i.e. Y - Z).

4.6 Base Colour

The inherent colour of either greasy or commercially scoured or carbonised wool after the removal of contaminants (e.g. grease, suint, dirt, vegetable matter) by laboratory cleaning processes.

4.7 "As is" Colour

The colour of the sample of commercially scoured or carbonised wool, as received at the laboratory. A conditioning test may be performed prior to colour measurement.

4.8 Delta Y

The difference between the Y tristimulus values determined from measurements of base and "as is" colour on the same global sample of commercially scoured wool.

5. Test Method

5.1 Apparatus

(a) A suitable spectrocolourimeter or spectrophotometer, capable of measuring in D65/10^{o} colour space and with a geometry of 45/0, with accessories, as described in Appendix A.

(b) A cell, to contain the test specimen, as described in Appendix A.

(c) A means of producing and maintaining the IWTO standard testing atmosphere as specified in IWTO-52.

(d) The apparatus required for test specimen preparation is detailed in Appendix B.

5.2 Preparation of Wool for Measurement

5.2.1 Sampling

If an IWTO Certificate is to be issued, the raw wool must be core-sampled and weighed in accordance with the current IWTO Core Test Regulations or the IWTO Condition Testing Regulations. For Scoured or Carbonised Wool the Test Method to be applied shall be either IWTO-19 or IWTO-33.

5.2.2 Test Specimen Preparation

The essential requirements for sample preparation for colour measurement are that the wool shall be cleaned of contaminants (e.g. grease, dirt, vegetable matter, etc.), well-blended, and conditioned. The procedures used in sample preparation should be such that the base colour of the wool is not altered.

Note 1: The Laboratory sample could have been prepared from either:

(a) Greasy wool or commercially scoured or carbonised wool (which has been subject to a laboratory scouring and drying process, and carded in accordance with Appendix B in order to determine the base colour); or

(b) Commercially scoured wool (which may have been subject to laboratory drying at 105°C for a condition test followed by carding in accordance with Appendix B in order to determine "as is" colour). Checks need to be undertaken to ensure that the procedures do not cause a change to the base colour of the wool.

Note 2: Caution, the colour of wool can be influenced by many factors during sample preparation, such as:

(a) *The quality of the water used for scouring samples*: this can be checked by scouring some samples in distilled or de-ionised water and comparing the colour measurements with samples that have been scoured normally. more than one scouring wash may be required including an intermediate drying step.

(b) *Drying*: prolonged drying at 105°C can yellow some wools, hence this should be avoided.

(c) *Air quality in the laboratory*: during drying and conditioning large volumes of air are passed over the wool. If the air contains a lot of dust, this can be trapped in the wool sample and may have an effect on the measured colour of the sample.

(d) *Shirley Analysing*: the perforated cage of the Shirley Analyser, and any surfaces that may come into contact with the wool should be checked to ensure that it does not impart any contaminants to the sample which affect the colour of the sample being tested.

(e) *Fluorescent lighting*: extended exposure to fluorescent lighting should be avoided as this changes the colour of the sample to be measured, particularly if the sample is close to the light source.

A procedure suitable for routine preparation is detailed in Appendix B.

5.3 Calibration of the Instrument

Spectrocolourimeter and spectrophotometers shall be calibrated in accordance with the procedures in Appendix D.

Note: Instruments shall be checked at least once every 8 hours of operation using a working calibration tile which has been measured against a certified tile as described in Appendix D. A check prior to the commencement of a series of measurements is considered to be acceptable laboratory practice.

5.4 Measurement

5.4.1 The procedure shall be as follows:

(a)After loading the test specimen into the cell, make 4 measurements of each tristimulus value according to either of the following methods:

(i) using one test specimen, measure each end, split the specimen and reassemble it to produce 2 new measurement faces and again measure each face; or

(ii)use 2 test specimens and measure 2 faces on each.

(b) Record the measurements.

5.4.2 Application of Range Checks in the Testing Process of Raw Wool

The critical range values are derived from the within laboratory standard deviation(12) and “Studentised range” values at the 99% level (Q*)(13). They are different for the various colour parameters and increase with the number of specimens measured as indicated below.

Critical Range Values for Colour Measurements of Raw Wool (99% Level of Confidence)

Parameter (units)	Within-lab Standard Deviation	Critical Range Number of Test Readings Q*			
		4 (4.40)	6 (4.76)	7 (4.88)	8 (4.99)
X	0.6263	2.8	3.0	3.1	3.1
Y	0.6577	2.9	3.1	3.2	3.3
Z	0.7157	3.1	3.4	3.5	3.6

The Range Check Procedures are presented as a flowchart in Figure 1 and as detailed procedures below.

(i) The testing process begins by reading 4 faces of the test sample.

(ii) If the range of the 4 readings is less than or equal to the critical value for 4 readings, the testing process is complete with 4 valid readings.

(iii) If the range of the 4 readings is greater than the critical range for 4 readings, measure another 2 faces either by splitting the test specimen as in Clause 5.4.1(a)(i) or by measuring both faces of a third test specimen.

(iv) If the range of the 6 readings is less than or equal to the critical value for 6 readings, the testing process is complete with 6 valid readings.

(v) If the range of the 6 readings is greater than the critical range for 6 readings, measure another 2 faces either by splitting the test specimen as in Clause 5.4.1(a)(i) or by measuring both faces of a fourth test specimen.

(vi) If the range of the 8 readings is less than or equal to the critical value for 8 readings, the testing process is complete with 8 valid readings.

(vii) If the range of the 8 readings is greater than the critical value for 8 readings, determine if one of the measurements is an outlier using the calculation procedure below[(5)]. Each measured parameter (X, Y and Z) is examined in order with the proviso that only one set of readings (i.e. X, Y and Z) can be deleted in Clause (c) with the possibility of a second set of readings being deleted in Clause (d). In the unlikely event that more than one test specimen is indicated as being an outlier for different parameters by Clauses (c) and (d) only the test specimen showing the greatest divergence is to be discarded.

(a) For the parameter exceeding the critical value, rank the 8 readings in ascending order of magnitude $n_1, n_2, ..., n_7, n_8$

(b) Calculate $R_1 = (n_8 - n_7) / (n_8 - n_2)$ and $R_2 = (n_2 - n_1) / (n_7 - n_1)$

(c) If $R_1 > 0.683$, discard all tristimulus values of n_8 and calculate the range of the 7 remaining readings. If the range of the 7 readings is less than or equal to the critical value of the 7 readings, the testing process is complete with 7 valid readings.

(d) If $R_2 > 0.683$, discard all tristimulus values of n_1 and calculate the range of the 7 remaining readings *(or 6 remaining readings if n_8 is also discarded in (c) above)*. If the range of the 7 measurements *(or 6 readings if n_1 and n_8 are both discarded)* is less than or equal to the critical value for the 7 readings *(or 6 readings if n_1 and n_8 are both discarded)*, the testing process is complete with 7 valid readings *(or 6 valid readings if n_1 and n_8 are both discarded)*.

(e) If, in either (c) or (d) above, the range of the 7 readings *(or 6 readings if n_1 and n_8 are both discarded in (c) and (d) above)*, still exceeds the critical value for 7 readings *(or 6 readings if n_1 and n_8 are both discarded in (c) and (d) above)*, retain all 8 readings and issue the mean result. Testing is complete with 8 valid readings. The wool is likely to be variable.

(f) If neither R_1 nor $R_2 > 0.683$, calculate the mean result of the 8 readings. The testing is complete with 8 valid readings. The wool is likely to be variable.

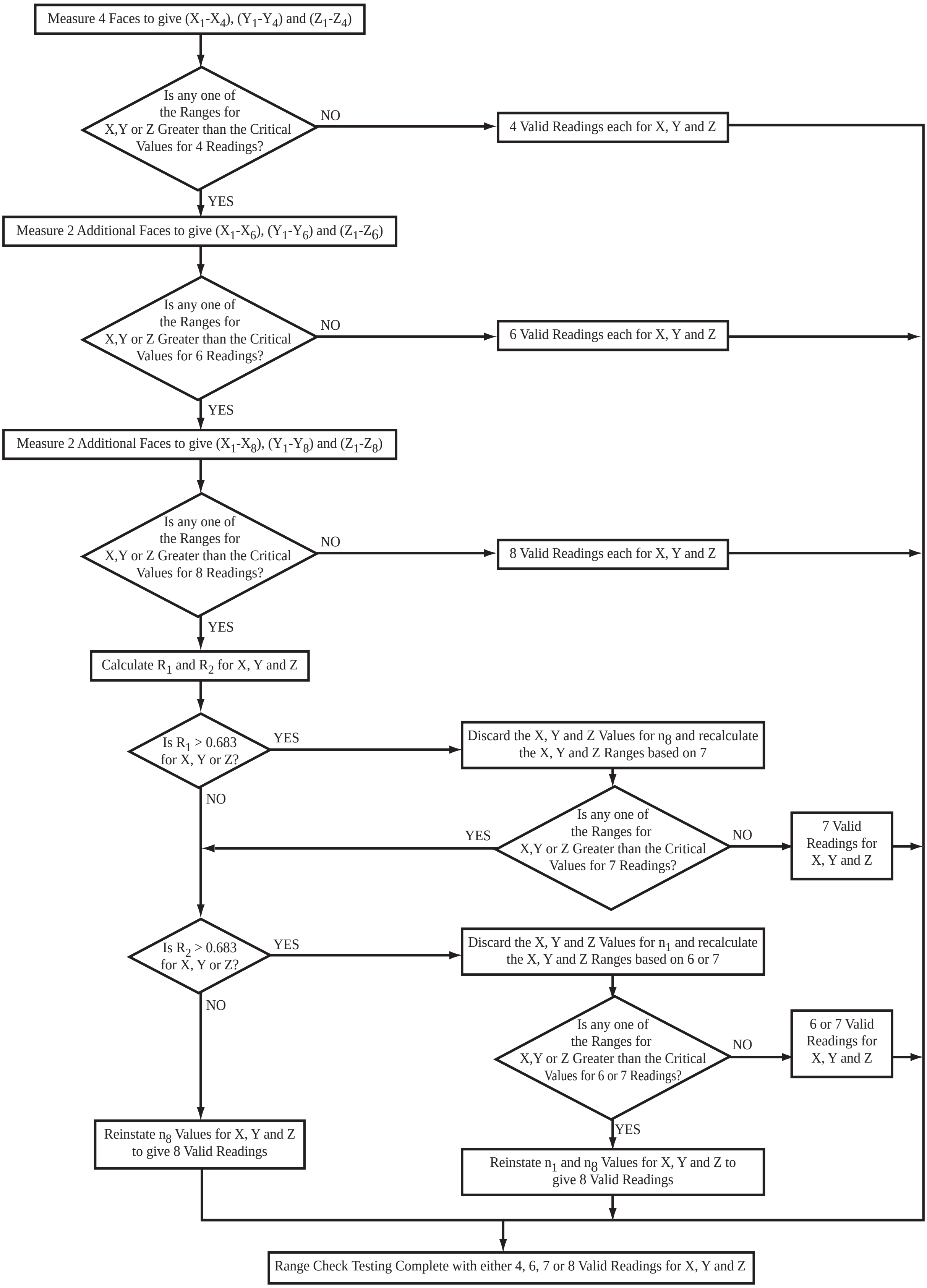

Figure 1: Procedures for Applying Range Checks

5.4.3 Calculating the Mean X, Y and Z
Calculate the mean tristimulus values of the cell glass (see Appendix C) and where necessary, correct the values so determined to tristimulus values in CIE colour space (see Appendix D).

Calculate the mean of the valid measurements of each tristimulus value to the nearest 0.1 unit.

5.4.4 Calculating Yellowness (Y-Z)
Calculate the difference between the mean Y value and the mean Z value to the nearest 0.1 unit.

6. Precision

6.1 Estimates of Components of Variance and 95% Confidence Limits

The between-laboratories (σ_b^2) and the within-laboratory (σ_w^2) components of variance[7], and the calculated precision limits, are presented in the following Table. The limits given are the 95% confidence limits.

Components of Variance and 95% Confidence Limits for Individual Tristimulus Measurements (X, Y, Z) and Average Yellowness Measurements (Y-Z)

	σ_w^2	σ_b^2	95% CL
X	0.46	0.63	2.06
Y	0.53	0.66	2.1
Z	0.67	0.75	2.2
Y-Z	0.08	0.13	0.9

7. Report

7.1 All Certificates shall show

(a) Number of bales sampled and tested.

(b) Clear identification of the test lot and test method used, including IWTO Test Code Number.

(c) Gross weight of test lot at time of coring.

(d) Declared tare.

(e) Weight of samples (if any) removed for appraisal after coring and weighing of the test lot.

(f) The nature of the raw wool if not greasy (e.g. scoured).

(g) For all colour test (base and “as is”), the following shall be reported in D65/10°:

(i) The mean tristimulus values X, Y and Z, each to the nearest 0.1 of a unit.

(ii) The mean value of the yellowness (Y-Z), to the nearest 0.1 of a unit.

7.2 All Certificates may show

For all colour tests (base and “as is”), the following may be reported in C/2°:

(i) The mean tristimulus values X, Y and Z, each to the nearest 0.1 of a unit.

(ii) The mean value of the yellowness (Y-Z), to the nearest 0.1 of a unit.

All values reported under Clause 7.2 shall be derived from one of the baremes in Appendix E of this Test Method. The reported values shall be annotated with a ‘w’ to indicate the Waring Blendor bareme has been used.

8. Bibliography

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APPENDIX A
THE MEASURING INSTRUMENT AND ITS ACCESSORIES

A.1 Measuring Instrument

A.1.1 A spectrocolourimeter or spectrophotometer based on the 45/0 geometry of the Commission Internationale de L'Eclairage is required. The instrument shall yield data relating to Illuminant D65 with 10^{o} Observer.

Not all 45/0 geometry instruments have been found to be suitable for issuing IWTO Certificates under this Test Method.

Appendix D lists instruments that have been shown to produce equivalent results and provides procedures to calibrate and validate instrument calibrations, including the procedures to be applied to correct for the presence of the glass in the wool sample holder. Procedures are also provided to verify the acceptability of new instruments against those listed in Appendix D.

A.2 Accessories

A.2.1 The accessories needed for the operation of an instrument calibrated to CERAM Certified Ceramic Tiles are as follows:

(a) a set of CERAM* IWTO Glossy Tiles with certified values for each tile

(b) a cream glossy tile.

A.3 Examples of Suitable Wool Cells

A3.1 Essential Requirements of all Wool Sample Holders

Provided that the same cell is used, both for the initial calibration and for the actual measurements, the design requirements are few. They are as follows:

(a) For Constant Density Cells, the wool shall be compressed behind glass to a packing density of 160 ± 5 kg/m^3. For Constant Pressure Cells, the wool shall be compressed behind glass to a minimum packing density of 160 kg/m^3.

(b) An "infinitely thick" layer of wool (i.e. that the light cannot penetrate through the sample) must be formed behind the glass. At the prescribed packing density this is achieved with a sample of minimum thickness of 8 mm.

(c) The glass should be colourless and polished on both sides.

(d) The glass thickness and the stand-off (the amount by which the cell glass is separated from the spectrophotometer or spectrocolourimeter port) should be kept small, preferably less than 3 mm each.

(e) Replacement glasses should be of the same refractive index and thickness as the original glass. If the glass is replaced, the set of CERAM tiles shall be measured behind glass. The results

*CERAM IWTO Glossy Tiles are available from: CERAM Research, Queens Road, Penkhull, Stoke on Trent, ST4 7LQ, United Kingdom. Fax + 44 (0) 1782 412331. E-mail: info@ceramres.co.uk.

from each tile must comply with the conditions of Clause D.3(ii). If D.3(ii) cannot be satisfied, a re-calibration, as detailed in Appendix D, is required.

(f) Replacement bulbs should be of similar specifications as the bulb used when calibrating the instrument. If the bulb is replaced, the set of CERAM tiles shall be measured behind glass. The results from each tile must comply with the conditions of Clause D.3(ii). If D.3(ii) cannot be satisfied, a re-calibration, as detailed in Appendix D, is required.

(g) The internal diameter of the cell shall be a minimum of 10 mm greater than the diameter of the instrument's diameter of measurement (port) so that there is no effect arising from reflections at the cell wall. The difference between the internal diameter of the cell and the instrument's port may be less than 10 mm, provided that the laboratory can show that the side of the cell is not detected.

(h) There shall be no Ultra-Violet (UV) filter placed in the illuminant path of the instrument.

(i) If the cell design is changed in any manner, equivalence shall be demonstrated using the procedures in Clause D.4.

A3.2 General Construction Details

Wool cells are of 2 types, constant density cells and constant pressure cells. The former requires a fixed mass of wool whereas the latter requires a minimum mass of wool. Figures A1, A2 and A3 are shown for guidance purposes, and dimensions are illustrative - exact dimensions will depend on the instrument port configuration, taken together with the Essential Requirements of A3.1.

Figure A1 shows a cell that can be used at constant density, where a fixed mass of wool is placed in a cylinder and the wool is compressed into a constant volume by an air cylinder that activates a plunger through a constant distance stroke.

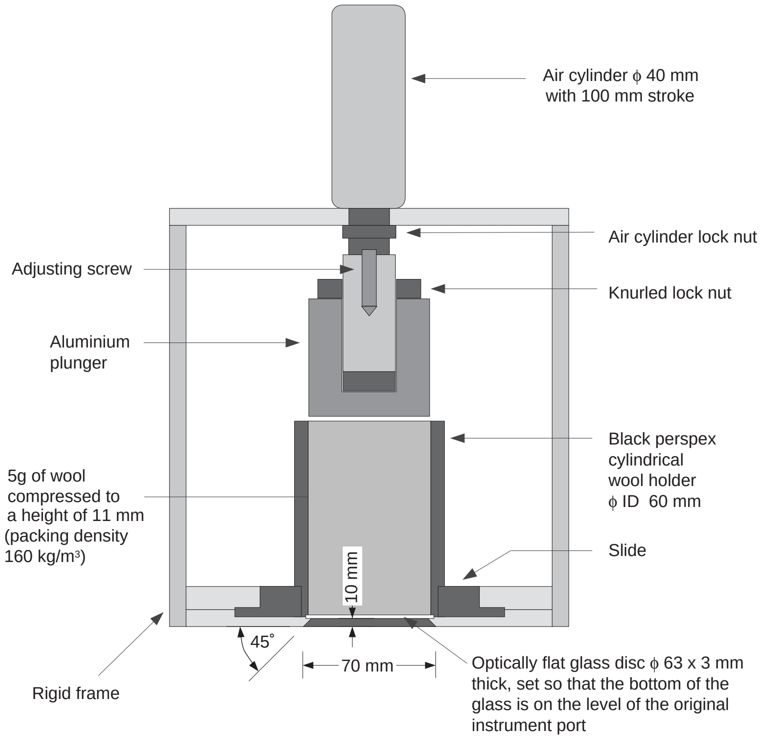

Figure A1: CONSTANT DENSITY CELL

Figure A2 is similar to Figure A1, but the air cylinder is fixed at a constant pressure when the plunger is activated. A minimum mass of wool can be measured for colour in this way.

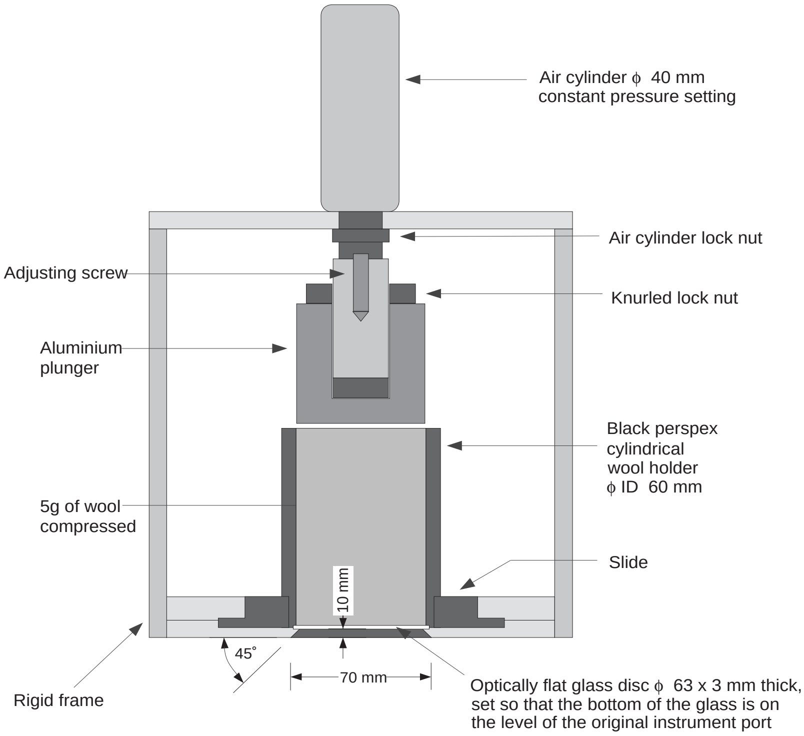

Figure A2: CONSTANT PRESSURE CELL

For a constant pressure cell, determine the pressure required to comply with specified density by using a minimum of 8 IH tops. Each top sample shall be cut into 2 cm lengths, Shirley analysed and conditioned. At lease 2 different known masses of specimens shall be used to cover the probable range of masses to be encountered in normal practice. For each of the masses, determine the pressures required on the first compression stroke (for example by measuring the air pressure supplied to the pneumatic cylinder) in order to achieve a wool density of 160 ± 5kg/m^3 in the cell. Calculate the average pressure for all determinations. This is the pressure that shall be used for the measurement. A means of monitoring the pressure shall be available.

Figure A3 shows a constant density cell where a fixed mass of wool is placed in a fixed volume cell

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Figure A3: CONSTANT DENSITY CELL

APPENDIX B
TEST SPECIMEN PREPARATION PROCEDURES

B.1 Introduction

The Mechanical Carding Method is suitable for the measurement of both brightness and yellowness. Modifications to the procedures are acceptable, provided that it can be shown that, using the procedures defined in IWTO-0 to establish equivalence, no alteration in the wool colour is induced thereby.

B.2 Mechanical Carding Method

The following method shall be considered the reference method against which any alternative method is to be compared for determining equivalence.

The procedure shall be as follows:

(a) For greasy wool, scour each of 2 approximately 150 g subsamples in a non-ionic detergent; scouring in alkaline conditions can lead to yellowing. The procedure given in IWTO-19 shall be followed, with the additional requirement that the sample be scoured twice, with intermediate hydroextraction (spin dry). The second scour of the sample, using reduced detergent load, is not a requirement of IWTO-19, but is required for this reference method against which alternative sample preparation methods must be compared.

(b) Remove excess moisture from the scoured subsample. This will normally be achieved by drying at 105°C as specified in IWTO-19; however, protracted drying at this temperature may cause yellowing and care should be taken to remove the wool from the dryer as soon as the dry state is attained.

(c) Draw equal quantities of wool from each subsample so as to provide, when combined, a laboratory sample of at least 20 g.

(d) Card the laboratory sample without excessive loss of wool to remove dust and vegetable matter. The procedure using a Shirley Analyser as given in IWTO-28 shall be followed.

(e) Bring the laboratory sample to equilibrium with the IWTO standard testing atmosphere as described in IWTO-52.

(f) Draw one or 2 test specimens (see Clause 5.4.1) of the required mass, removing any residual vegetable matter or other visible contaminant with tweezers.

(g) For a constant volume cell weigh out test specimens to give a packing density of 160 ± 5 kg/m^3.

Note: Remove, with tweezers, any vegetable matter or other non-wool material likely to affect the colour measurement. Draw test specimens of the required mass from the laboratory sample. Weigh the test specimens to 0.1 g for the constant volume cell.

APPENDIX C
TRANSFORMING VALUES TO CIE UNITS AND DETERMINATION OF CELL GLASS CORRECTIONS

When a colour measuring instrument is calibrated to a Certified Reference Standard (i.e. a ceramic tile with assigned values traceable to the National Physical Laboratory (NPL) in the United Kingdom), the glass correction factors must be determined as set out in Appendix D. These corrections are applied to the measured tristimulus values prior to them being reported.

All instruments use a 2-point calibration (i.e. a black reference point and a white reference point). The calibration software supplied with the instrument will prompt the user to place the appropriate standard over the instrument port during its calibration procedure. It is preferable that the CERAM certified standards be used. However, some manufacturers have built into their instrument software a calibration that prevents the use of any standard other than the one they have supplied. The data from such instruments must be transformed externally to meet the requirements of this Test Method. Procedures to be used in both situations are provided below.

C.1 Transforming Values into CIE Units

C1.1 Instruments that use the CERAM standards for calibration

No corrections are required for instruments that use the CERAM standards for calibration.

C1.2 Instruments that use only the manufacturer's standards

For instruments which use the manufacturer's standards for calibration, a correction to CIE units must be applied. The following procedures are applicable to instruments that can only be calibrated with the instrument manufacturer's standards:

(a) Select the D65/10° option for measurements.

(b) Calibrate the instrument using the manufacturer's procedures and standards against the port of the instrument.

(c) Measure the X, Y and Z values of each of the CERAM certified tiles directly against the instrument port 4 times, rotating the tile through 90° between each measurement.

(d) Calculate the average "against-port" X, Y and Z values for each tile.

(e) Tabulate the certified value of each CERAM tile and its "against-port" value.

Note: The CERAM black tile is excluded because of measurement difficulties with some instruments. In situations where the manufacturer's black reference is not as black as the CERAM black, and the software does not permit the display of negative numbers, erroneous values will be displayed.

(f) Regress the Certified Values against the measured "against-port" (MAP) values of the tiles for each of X, Y and Z. Note that these equations are not to be forced through zero. The general forms of these equations are:

$$X_{CIE} = a_1 + b_1 X_{MAP}$$

$$Y_{CIE} = a_2 + b_2 Y_{MAP}$$

$$Z_{CIE} = a_3 + b_3 Z_{MAP}$$

where:

X_{CIE} = Certified X Value for Tile

Y_{CIE} = Certified Y Value for Tile

Z_{CIE} = Certified Z Value for Tile

X_{MAP}= X value measured against the instrument port

Y_{MAP}= Y value measured against the instrument port

Z_{MAP}= Z value measured against the instrument port

a_1 = regression constant for X

a_2 = regression constant for Y

a_3 = regression constant for Z

b_1 = regression slope for X

b_2 = regression slope for Y

b_3 = regression slope for Z

C.2 Determination of Cell Glass Corrections

The following procedures are applicable to all instruments that have been calibrated against the instrument port with a reference colour standard. As the wool sample is measured in a sample holder with a glass window, rather than directly against the port, the effect of the sample holder must be determined. It is then used to correct any measurements made in that sample holder. The procedures are as follows:

(a) Ensure the D65/10° instrument option has been selected.

(b) Measure the CERAM tiles against the port of the instrument as follows:

(i) Calibrate the instrument using the manufacturer's procedures either with the CERAM black and white standards or the manufacturer's standards against the port of the instrument.

(ii) Measure the X, Y and Z values of each of the CERAM certified tiles directly against the instrument port 4 times, rotating the tile through 90° between each measurement.

(iii) Calculate the average "against-port" X, Y and Z values for each tile.

(c) Measure the CERAM tiles behind glass as follows:

(i) Place the glass of the cell to be used for subsequent measurement over the instrument port in the same position that it will be used for measurement.

(ii) Calibrate the instrument using the manufacturer's procedures either with the CERAM black and white standards or the manufacturer's standards on top of the glass against the port of the instrument (i.e. behind the glass as far as the light source is concerned).

(iii) Measure the X, Y and Z values of each of the CERAM certified tiles behind the glass 4 times, rotating the tile through 90° between each measurement.

(iv) Calculate the average "behind-glass" X, Y and Z values for each tile.

(d) Calculate the glass correction equations for X, Y and Z separately (as per the procedures in C.3).

C.3 Example Calculations for Transforming Measured Values into CIE Units and Determining the Glass Corrections

Below are example values obtained after calibration and measurement of tiles against the instrument port and behind the glass. An example is also shown for transforming the measured values into CIE units.

Note that the black is excluded because of measurement difficulties with some instruments.

C.3.1 Example Calculations for Transforming Measured Values into CIE Units

Equations for transforming measured values to CIE units can be calculated as shown below:

	Certified Values			Measured Values (Against-Port)		
Tile	X_{CIE}	Y_{CIE}	Z_{CIE}	X_{MAP}	Y_{MAP}	Z_{MAP}
White	83.57	88.28	92.86	84.79	89.54	94.23
80% Grey	75.97	80.26	84.94	77.07	81.36	86.22
70% Grey	66.68	70.50	75.24	67.83	71.69	76.52
Pale Grey	58.17	61.49	65.55	59.26	62.59	66.63
50% Grey	47.58	50.31	53.83	48.50	51.25	54.98
40% Grey	37.85	40.08	42.80	38.55	40.81	43.59
33% Grey	32.63	34.32	36.77	33.34	35.05	37.56
Mid Grey	23.61	24.93	26.46	24.21	25.54	27.01
Deep Grey	4.50	4.73	4.83	4.62	4.84	4.93
Wool 1	66.85	70.35	60.52	68.06	71.62	61.70
Wool 2	57.39	59.73	45.28	58.47	60.87	46.13

Using Clause C.1.2, the following formula are produced:

$$X_{CIE} = -0.20909 + 0.986563 * X_{MAP}$$

$$Y_{CIE} = -0.21372 + 0.986845 * Y_{MAP}$$

$$Z_{CIE} = -0.19185 + 0.98618 * Z_{MAP}$$

The measured values can then be transformed into CIE units and compared against the certified CERAM tiles:

:

	Certified Values			Transformed Values)			Differences (Transformed-Certified)		
Tile	X_{CIE}	Y_{CIE}	Z_{CIE}	X_{CIE}	Y_{CIE}	Z_{CIE}	X	Y	Z
White	83.57	88.28	92.86	83.44	88.15	92.73	- 0.13	- 0.13	- 0.13
80% Grey	75.97	80.26	84.94	75.83	80.08	84.83	- 0.14	- 0.18	- 0.11
70% Grey	66.68	70.50	75.24	66.71	70.54	75.27	0.03	0.04	0.03
Pale Grey	58.17	61.49	65.55	58.25	61.55	65.51	0.08	0.06	- 0.04
50% Grey	47.58	50.31	53.83	47.64	50.36	54.03	0.06	0.05	0.20
40% Grey	37.85	40.08	42.80	37.82	40.06	42.79	- 0.03	- 0.02	- 0.01
33% Grey	32.63	34.32	36.77	32.68	34.38	36.85	0.05	0.06	0.08
Mid Grey	23.61	24.93	26.46	23.67	24.99	26.44	0.06	0.06	- 0.02
Deep Grey	4.50	4.73	4.83	4.34	4.56	4.67	- 0.16	- 0.17	- 0.16
Wool 1	66.85	70.35	60.52	66.93	70.46	60.66	0.08	0.11	0.14
Wool 2	57.39	59.73	45.28	57.47	59.86	45.30	0.08	0.13	0.02

C.3.2 Example Calculations for Determining the Glass Corrections

Glass corrections can be calculated by following Clause C.2 as shown below:

CERAM Tile	Against-port (MAP)			Behind-glass (MBG)		
	X_{MAP}	Y_{MAP}	Z_{MAP}	X_{MBG}	Y_{MBG}	Z_{MBG}
White	84.79	89.54	94.23	84.87	89.60	94.07
80% Grey	77.07	81.36	86.22	76.33	80.55	85.21
70% Grey	67.83	71.69	76.52	66.46	70.22	74.88
Pale Grey	59.26	62.59	66.63	57.45	60.66	64.48
50% Grey	48.50	51.25	54.98	46.42	49.05	52.53
40% Grey	38.55	40.81	43.59	36.46	38.58	41.15
33% Grey	33.34	35.05	37.56	31.31	32.91	35.23
Mid Grey	24.21	25.54	27.01	22.50	23.73	25.04
Deep Grey	4.62	4.84	4.93	4.17	4.38	4.43
Wool 1	68.06	71.62	61.70	66.68	70.09	59.34
Wool 2	58.47	60.87	46.13	56.73	58.92	43.68

Research[11] has shown that the correction factors will take the general form of a quadratic equation, as described below:

For X, $C = c_1(x^2 - T_1x)$ For Y, $C = c_2(x^2 - T_2x)$ For Z, $C = c_3(x^2 - T_3x)$

where:

C = correction factor

c_1 = constant value for X calculations

c_2 = constant value for Y calculations

c_3 = constant value for Z calculations

T_1 = assigned (or certified) X value of white calibration standard

T_2 = assigned (or certified) Y value of white calibration standard

T_3 = assigned (or certified) Z value of white calibration standard

x = relevant "behind-glass" tristimulus value (i.e. X, Y or Z) of material being measured.

Note: Where a cream glossy tile is used to transform the CERAM calibration to itself, the assigned values for this tile shall be used for T_1, T_2 and T_3.

For this example, the assigned values of the calibration standard were:

X = 82.40 Y = 87.25 Z = 93.80

The following steps must be carried out to calculate the glass corrections for each of X, Y and Z.

(a) For each tile, the difference (D) between the "behind-glass" (MBG) and "against-port" (MAP) reading must be calculated for X, Y and Z. The "behind-glass" value must be subtracted from the "against-port" value. In this example, the difference for X on the white CERAM tile is:

$$D = 84.79 - 84.87$$
$$= -0.08$$

Thus all the differences for this example are as follows:

(b) For each tile, calculate (x^2-T_nx) for each of X, Y and Z. In this example, (x^2-T_nx) for X on the white CERAM tile is:

$$(x^2-T_1x) = (84.87)^2 - (82.40 \text{ x } 84.87)$$
$$= 209.85$$

:

CERAM Tile	Difference (D) (Against port - Behind glass)		
	ΔX	ΔY	ΔZ
White	-0.08	-0.05	-0.16
80% Grey	0.74	0.81	1.01
70% Grey	1.37	1.47	1.64
Pale Grey	1.81	1.93	2.15
50% Grey	2.08	2.20	2.45
40% Grey	2.09	2.23	2.44
33% Grey	2.03	2.14	2.34
Mid Grey	1.71	1.81	1.97
Deep Grey	0.44	0.46	0.50
Wool 1	1.38	1.53	2.36
Wool 2	1.74	1.96	2.45

Thus, the remainder of the (x^2-T_nx) results are as follows:

CERAM Tile	x^2-T_1x		
	X	Y	Z
White	209.85	210.10	24.93
80% Grey	-463.15	-540.05	-731.95
70% Grey	-1059.25	-1195.71	-1416.59
Pale Grey	-1433.46	-1612.86	-1890.64
50% Grey	-1670.19	-1873.68	-2167.94
40% Grey	-1674.95	-1877.69	-2166.55
33% Grey	-1599.68	-1788.33	-2063.30
Mid Grey	-1347.84	-1507.43	-1721.53
Deep Grey	-326.40	-363.17	-395.48
Wool 1	-1048.34	-1202.88	-2044.92
Wool 2	-1456.34	-1669.28	-2189.24

(c) For each of X, Y and Z, regress the differences generated in (a) against the corresponding values generated in (b). It is essential to force the regression through zero. This will determine the constant values (i.e. c_1, c_2 and c_3) for each of X, Y and Z.

For this example, the constants were:

For X, c_1 = -0.0012580425 For Y, c_2 = -0.0011978315 For Z, c_3 = -0.0011396470

(d) For subsequent measurements using the tile calibration, a glass correction for each of X, Y and Z should be calculated and added to the "behind-glass" measurement of the wool samples. For example, if a sample had a Y_{MBG} value of 60.45 (performed "behind-glass"), the following correction would be applied:

$$C = -0.0011978315 \times ([60.45]^2 - [87.25 \times 60.45])$$

$$= 1.94$$

Thus the corrected result would be:

$$Y_{MAP} = Y_{MBG} + 1.94$$

$$Y_{MAP} = 60.45 + 1.94$$

$$= 62.39$$

If a further correction to CIE D65/10° colour space was required, this would use the regression determined in Clause C.3.1 as follows:

$Y_{CIE} = 0.986845\ Y_{MAP} - 0.21372$

$Y_{CIE} = (0.986845 * 62.39) - 0.21372$

$= 61.36$

Care should be taken at all stages not to truncate any results as rounding errors may occur.

APPENDIX D
CALIBRATION WITH CERTIFIED CERAM TILES

D.1 Introduction

All colour measuring instruments are standardised to 2 reference points. These are typically, a black tile and a white tile, that have values assigned to them by either the manufacturer of the instrument or an independent certifier of colour reference standards. It has also been recognised that the 2 primary suppliers (one in Canada and one in Germany) of the ultimate traceability for white reference standards are different from one another. For this reason this Test Method is specific in its choice to maintain traceability to the German system through the National Physical Laboratory (NPL) in the United Kingdom. The objective of the procedures described in this Appendix is to ensure that all instruments can be calibrated to the one colour scale.

Originally the calibration method was developed using wool with assigned vales in the Illuminant C (with an Observer angle of 2^o) colour space[8]. At the IWTO Meeting, Nice, December 1997, a decision was taken to move to the colour space more commonly used by dyers (i.e. Illuminant D65 with an Observer angle of 10^o).

This Appendix details the procedures to be used to calibrate instruments using Certified CERAM tiles. Validation procedures, firstly against the instrument port (i.e. without any glass in the optical path) and secondly "Behind-glass" (i.e. with the glass to be used for measurements in the optical path and with the application of the glass corrections applied) are included.

It has been demonstrated that some instruments can meet the above validation requirements and still introduce consistent biases against other instruments when measuring the same wool sample[14]. An industry decision was taken in December 2002 by IWTO to use as a base those instruments that were commonly used in Raw Wool testing laboratories at that time that showed agreement, as the base for deciding the acceptability or otherwise of any future instruments. The instruments found to produce harmonised results were:

- The BYK Gardner Color Machine (TCM) using a 32 mm port;
- the BYK Gardner Color View (TCV); and
- the Hunterlab Miniscan XE.

This Appendix also includes procedures for demonstrating the equivalence of alternative instruments to those listed above to enable new technology to be adopted as it becomes commercially available.

D.2 Certified Tile Calibration (Illuminant D65/10^o Observer)

D2.1 Measurement of the CERAM calibration tiles against the instrument port

Each of the CERAM tiles (excluding the black tile) must be measured against the instrument port. This is performed as follows:

(a) Measure the X, Y and Z values of each of the CERAM certified tiles against the instrument port 4 times, rotating the tile through 90^o between each measurement.

(b) Calculate the average "against-port" X, Y and Z values for each tile.

(c) Where necessary, transform the measured values into CIE values using the equations derived in Clause C.1.2.

D2.2 Measurement of the CERAM calibration tiles behind glass

After measuring the CERAM tiles against the instrument port, the same set of tiles must be measured behind the glass window which separates the instrument port from the sample holder. This is performed as follows:

Calibrate the instrument using the manufacturer's procedures and the CERAM black and white standards behind the glass (after correcting values for the effects of the glass cell and, where necessary, correcting values to CIE units).

Measure the X, Y and Z values of each of the CERAM certified tiles behind the glass 4 times, rotating the tile through 90^o between each measurement.

Calculate the average "behind-glass" X, Y and Z values for each tile.

Correct all values for the effects of the glass cell (using the equations derived in Appendix C.2) and, where necessary, transform the measured values into CIE values using the equations derived in Clause C.1.2.

D.3 Validation of Against-Port and Behind-Glass Measurements

Acceptability of the CIE Tile Calibration

A CIE calibration is deemed to be satisfactory if:

(i) "Against-port" measurements (after correcting values to CIE units, where necessary) are less than 0.5 units from their respective certified CERAM value of each individual tile; and

(ii) "Behind-glass" measurements (after correcting values to CIE units) are less than 0.75 units from their respective certified CERAM value for each individual tile and the average differences over the 11 tiles (i.e. black excluded) are less than 0.4 for X, Y and Z and 0.2 for Y - Z.

If the above criteria are not satisfied, check to ensure that the CERAM tiles are clean and undamaged. Repeat the calibration process until Clauses D.3 (i) and D.3 (ii) are satisfied.

D.4 Determining Equivalence of Alternative Instruments

Prior to the use of any instrument other than the BYK Gardner TCM, the BYK Gardner TCV and the Hunterlab Miniscan XE, the results from the instrument must be shown to be equivalent to the named instruments using the procedures set down in IWTO-0.

A minimum of 75 different samples, exhibiting a range in Y from at least 50 - 72 (in D65/10^o colour space) and a range in Y - Z (in D65/10^o colour space) from at least 7 - 17, shall be measured on the new instrument and one of those nominated above. The results for the individual sample averages for the 3 tristimulus measurements for X, Y and Z shall be compared to determine the average difference and the statistical significance or otherwise of any level dependent bias as detailed in IWTO-0.

If the level dependent bias is not statistically significant at the 5% probability level and the average difference is less than 0.5 for each of the 3 tristimulus values and Y - Z, the alternative instrument is acceptable for use under this Test Method.

It is the responsibility of the laboratory using the alternative instrument to either:

- Present a technical report to the IWTO Technology and Standards Committee detailing the comparative data and requesting to have the name of the new instrument included in the Test Method; or

- Retain the data and analysis which met the requirements above for a period at least seven (7) years for verification by the National Accrediting Body during its audits.

APPENDIX E
BAREMES FOR CONVERTING FROM THE MEASURED D65/10º TO THE OLD C/2º COLOUR SPACE

It is envisaged that the following baremes will have a short lifespan (effective to 1 July 2004) to enable commercial traders to adjust to the new D65/10º colour space.

Due to the historical situation in New Zealand, which has a large database of certified colour measurements based on the Waring Blendor Preparation route, 2 baremes are provided:

- a Measurement-only bareme; and
- a Waring Blendor preparation bareme.

E.1 Measurement Bareme

The baremes that shall be used for measurement only are detailed by the following formulas.

The tristimulus values measured in D65/10º colour space and using the preparation system detailed in Appendix B will be different to measurements produced in the past using C/2º colour space and the reference wool calibration method. For guidance, the following conversion formulae(6) may be used:

$$X_W = -3.6935 + 0.9834\ X_t$$

$$Y_W = -1.9252 + 0.9298\ Y_t$$

$$Z_W = -1.4508 + 1.0405\ Z_t$$

$$(Y\text{-}Z)_W = Y_W - Z_W$$

Where: X_W, Y_W and Z_W are the tristimulus values equivalent to those which would have been measured by an instrument calibrated to reference wool (based on Illuminant C and 2º Observer angle); and

X_t, Y_t and Z_t are the tristimulus values measured by an instrument calibrated to certified tiles (based on Illuminant D65 and 10º Observer angle) and corrected for any glass effects. The samples having been prepared using the procedures detailed in this Test Method.

E.2 Waring Blendor Bareme

The baremes that shall be used for converting to Waring Blendor C/2º values are detailed by the following formulas.

$$(WB)X_W = -3.3059 + 1.0222\ X_t$$

$$(WB)Y_W = -0.8881 + 0.9562\ Y_t$$

$$(WB)Z_W = -2.6089 + 1.1415\ Z_t$$

$$(WB)(Y\text{-}Z)_W = (WB)Y_W - (WB)Z_W$$

Where: $(WB)X_W$, $(WB)Y_W$ and $(WB)Z_W$ are the tristimulus values equivalent to those which would have been measured by an instrument calibrated to reference wool (based on Illuminant C and 2^o Observer angle); and X_t, Y_t and Z_t are the tristimulus values measured by an instrument calibrated to certified tiles (based on Illuminant D65 and 10^o Observer angle) and corrected for any glass effects. The samples having been prepared using the procedures detailed in this Test Method.

METHOD FOR THE MEASUREMENT OF COLOUR OF RAW WOOL

Amendments issued since publication:

Date of issue	Text affected
Amendments to IWTO-56-00 approved Barcelona, May 2002. Full reprint: IWTO-56-02 issued September 2002.	Additions to range checking procedures in Clause 5.4.2. Addition of Figure 1 to Clause 5.4.2.
Revision of IWTO-56-02 approved Nice, November 2002. **Full reprint: IWTO-56-03 issued April 2003**	**Removal of the following options:** **to make measurements in C/2 colour space;** **to calibrate with Reference wool;** **to use Waring Blendor.** **Additional editing of Specification.**

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